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Preparation and Applications of Flavor Concentrates From Deciduous Fruits

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One factor which has contributed to advancing our knowledge of flavors, especially volatile fruit flavors, has been the development of effective means for recovering aromas in concentrated unaltered form. This has been the work of many individuals and groups. My own concern will be with the research carried out at the Department of Agriculture's Eastern Regional Research Laboratory in Philadelphia, where the first commercially practical process for aroma recovery was developed about a decade ago and where research in this field has continued to the present time.

Our work in the field of flavor recovery was undertaken to produce concentrated fruit juices without losing their aroma. This was finally accomplished by stripping the aroma from the juice under conditions to avoid flavor change, simultaneously fractionating the aroma to an essence, and restoring the essence to the vacuum concentrated juice. This is novel only in the means used to accomplish it. Since this is a flavor conference, my primary emphasis will be with regard to the essence recovery process,[b] its evolution during the past 10 years, as well as its likely potentialities for improving food flavors.

Early investigators concentrated the fresh fruit juice under vacuum and attempted to recover the volatile flavors by condensing them at low temperature; perhaps redistilling the condensate to further concentrate flavor. Although logical from the viewpoint of avoiding flavor damage, the use of vacuum has one important drawback. All fruit juices contain dissolved air or other gases, and in any flavor recovery system they must be vented since they do not condense. Unless special precautions are taken, such as I will discuss later, this gas leaves the system carrying some aroma with it. The loss of volatiles in the vent gas is roughly in inverse proportion to the absolute pressure at which the system is operated. For example, when the gases are vented from a system under a 27-inch vacuum (3-in. absolute pressure) the losses are more than 10 times as great as when venting at atmospheric pressure. This is true even when the gases are in both cases

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[b] The term "essence" as used here refers to a distilled aqueous solution of aromas more concentrated than in the parent fruit.

chilled and inleakage of air is neglected. In practice there would e some inleakage of air which would further exaggerate this difference. This factor of vent gas loss can be serious when the amount of vent gas is high as a consequence, for example, of a slight fermentation of the juice. Vent gas losses also are magnified with increasing concentration of the distillate and, of course, with increase in volatility of the product. It is always the top notes which are first lost in such a system, throwing the flavor out of balance.

Early Design

With these objections to vacuum in mind, Milleville and coworkers at the Eastern Laboratory in 1944 designed a unit to operate at atmospheric pressure and above.[c] I think I can safely say that with this apparatus they were the first to recover apple essence in concentrated, substantially unaltered form. This enabled us to make a full-flavor apple juice concentrate which gave a delightfully fresh flavor on reconstitution. Figure 1 shows the arrangement of this apparatus. I want to devote a few minutes to it because it was the progenitor of many flavor recovery units of improved design. The juice, in this case apple juice, was fed at a constant rate to a super-heater wherein its temperature was raised in about 3 seconds to 320° F. Flashing the heated juice to atmospheric pressure was sufficient to vaporize about 10 percent. revious investigators had shown that all the volatiles in apple uice appeared in the first 10 percent evaporated. The liquid and vapor were separated in a vapor-liquid separator, the stripped juice passing to a vacuum evaporator for rapid cooling and concentration. The vapors, now concentrated about 10-fold, passed to a fractionating column for further concentration to the desired degree. Usually this was 100-fold, that is, the aroma was collected in a volume corresponding to 1/100 that of the starting juice. The vapors issuing from the top of the column were condensed, a part being returned as reflux and the remainder being drawn off at a metered rate with respect to the juice feed rate. The product was apple essence which, when restored to the concentrated stripped juice, gave the full-flavor product mentioned above.

Our success with apples suggested that volatile flavor recovery might have much broader applications. However, although the

[c] "Recovery and Utilization of Natural Apple Flavors," by Howard P. Milleville and Roderick K. Eskew, AIC–63, September 1944. Also in the Fruit Products Journal and American Food Manufacturer, vol. 26, pp. 48–51, October 1944.

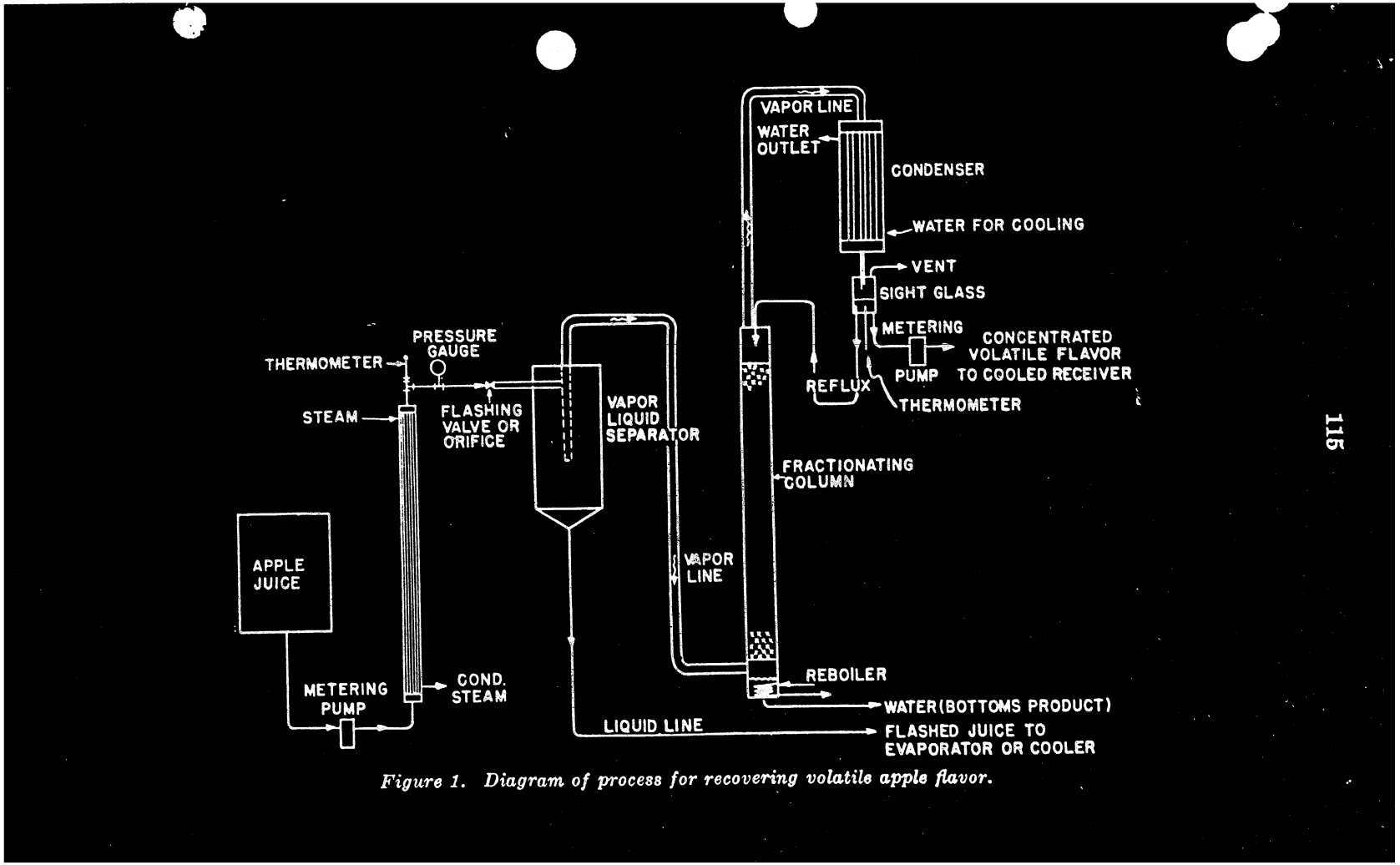

Figure 1. Diagram of process for recovering volatile apple flavor.

basic principles of the process were found to be applicable to the juice of many other fruits, the early apparatus designs gave mediocre results in most cases. Even when a rapid evaporator was substituted for a super-heater and when the vent gases leaving the system were thoroughly chilled with the volatile fruit concentrate before being vented, the apparatus was still unsatisfactory for some fruit juices. In some cases the juice flavor was altered by the heat incident to vaporizing the volatiles, and in others the desirable top notes were lost.

Improved Design Extends Utility

The arrangement shown in figure 2 is much more versatile than the earlier designs and can be used with all but the most heat-sensitive juices. The juice is fed by a positive delivery pump at a constant rate to an atmospheric preheater where its temperature is brought approximately to its boiling point. It then passes into the vaporizer where the amount required for aroma release is vaporized. This varies widely depending upon the fruit. For apples it is 8 or 10 percent and, for grape juice, 40 or more percent. This separation of the heating and vaporization steps permits accomplishing the aroma release with the minimum heat effect. The preheater consists of a small-diameter tube through which the juice travels at such velocity that it is in turbulent flow and, in consequence, is rapidly and uniformly heated. If the velocity in this tube is not less than 20 feet a second, fouling of the tube walls, which resulted with apple juice in earlier designs, is eliminated. In this arrangement the juice can be heated, the desired percentage vaporized, and the juice again cooled by flashing into vacuum in a total time of about 3 seconds. This is very much faster, and hence there is less heat damage than in earlier designs.

The aroma-bearing vapors enter a fractionating column where they are concentrated. They then are condensed and a portion is drawn off as product. You will note, however, that there is quite a different method of handling the non-condensable gases before they are vented. Instead of releasing them directly to the atmosphere or chilling with essence as was done in earlier designs, they are countercurrently scrubbed with cold water. In this case the scrubbing liquid is column bottoms. This means they leave the system in equilibrium with water and hence are free of aroma. In earlier designs when the gases were cooled with essence they were vented in equilibrium with aroma-rich liquid.

The scrubbing liquid, containing whatever top notes it has picked up from the vent gases, is returned to the column. Because of this liquid feed, we require a stripping section in the column where formerly it was not necessary. We have used this arrangement with good success in making essences from apples, grapes, cherries, blackberries, blueberries, strawberries, and other fruits. It should be applicable to many other juices and will permit the making of high-fold essences with good recovery because of the system used in treating the vent gas.

Vacuum Operation Required for Some Juices

There are juices which are too heat sensitive to be handled at atmospheric pressure, notably orange juice. The whole system then must be operated under vacuum. This is not merely a matter of attaching a source of vacuum to a unit designed for atmospheric use. Although the basic steps in the process of flavor recovery remain the same, the apparatus must be modified chiefly as regards the relative size of its component parts. For instance, if the system is to operate at the same juice rate and under 3 inches absolute pressure, the fractionating column and some other vapor handling parts must be enlarged to accommodate about 10 times the volume of vapor that they were required to handle at atmospheric pressure.

With vacuum operation we must, of course, be especially careful to avoid aroma loss in the vent gas. This can be prevented by scrubbing the gas with chilled column bottoms as shown in figure

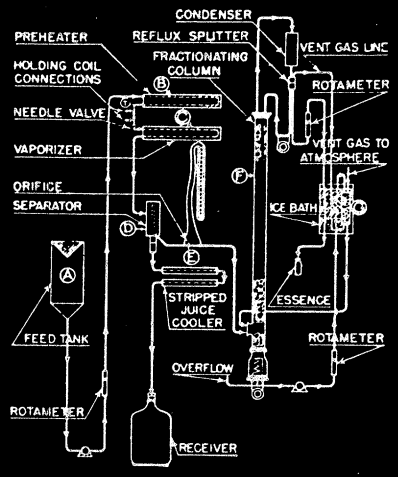

Figure 2. Improved apparatus for reducing vent gas losses.

2 although, of course, the scrubbing system must be enlarged to accommodate the increased volume of gas at the lower pressure. We built such a unit on a pilot-plant scale. It was used at our Bureau's Winter Haven, Florida, laboratory and could strip and concentrate the aroma of orange juice without heat damage to the juice. However, there is some question as to the practical value of the essence so recovered.

Single Pass Atmospheric Concentration

It was stated earlier that 40 or more percent vaporization is necessary to release aroma from Concord grape juice. Methylanthranilate, the component that characterizes Concord grapes, is tenaciously retained in the juice. A residual aroma still can be detected after 60 or more percent of the liquid has been vaporized. This adversity can be turned to advantage by combining aroma release and juice concentration into a single operation.

Naturally, if a high density concentrate, for example 7-fold, is to be made, the juice first must be depectinized. This can be done conveniently simultaneously with detartration.

Figure 3 illustrates how such an apparatus is used. The detartrated, depectinized juice is preheated and passes to the vapor-

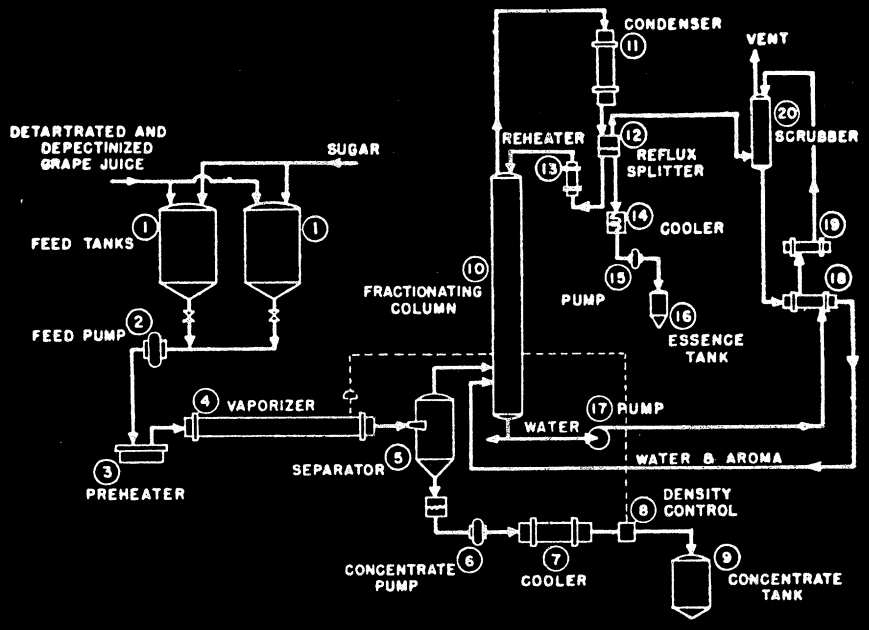

Figure 3. Single pass atmospheric concentration of grape juice.

izer in the conventional manner. At this point, however, instead of vaporizing 40 or 50 percent all the liquid is driven off necessary to concentrate to about 75° Brix. The essence recovery apparatus is substantially the same as that we have already described but somewhat larger in proportion to the vaporizer because of the greatly increased amount of vapor to be handled. Any methylanthanilate remaining in the concentrate will appear in the finished product, and if the recovered essence is restored the product will be approximately full flavor. I qualified that term "full-flavor" deliberately, for our tests have shown that less than half of the methylanthranilate which goes to the fractionating column actually appears in the essence. The remainder is lost in column bottoms. Fortunately a little bit goes a long way, so that this much loss is not serious from a practical standpoint. We recognize, however, that there is a need to improve the system for recovering Concord grape essence and are now studying the problem.

Heat Effect Sometimes Desirable

In contrast to juices which must be handled under vacuum, there are others in which the desired flavor can be intensified by deliberate heating. Montmorency and Morello cherry juices are in this category. We have found that if Montmorency cherry juice is heated for 1¼ minutes at 230° F. it will develop more aroma. If the aroma is then stripped and restored to the juice concentrate, the product will have much more cherry flavor than if made from unheated juice. This isn't very surprising for we all know good cherry pie has more of what we call cherry flavor than does the raw fruit itself.

Concord grape juice also benefits flavor-wise from some degree of heat. Flavor recovery studies made at various stages of conventional manufacture show a progressive flavor enhancement up to and through the aging process for tartrate settling. Undoubtedly some other fruits would be better for heat treatment. We are all familiar with flavor development by the roasting of coffee beans, the fermentation of vanilla pods, the aging of whisky, and the cooking of many foods. I wonder if we have yet exhausted the possibilities of flavor enhancement (and I mean either modification or intensification) in the case of fruits. I am told that in Europe volatile fruit flavors may be altered deliberately during distillation by a catalysis which favors ester formation. Thus, the latent aroma of unripened fruit may be said to develop during

recovery to a semblance at least of that found in the sun-ripened product.

I have talked a great deal about fruit juices, for most of our experience has been in this field. To an ever increasing extent, juices are being sold in the form of concentrates. If these concentrates are made by vacuum evaporation they will not yield juices of satisfactory quality unless the volatiles are restored. In the case of citrus juices, this is of course done by adding some fresh cutback juice to the concentrate. This has proven satisfactory because the chief flavoring constituent here is in the peel oil contained in the cutback juice. However, if fresh flavor is to be had in high-density (e.g. 7-fold) concentrates made from non-citrus juices, essence must be restored. If then the volatiles must be restored to the juice concentrates, how will they ever appear on the market as essences for use by manufacturers of extracts, beverages, candy, or other food products?

Volatile Fruit Concentrates from Preserve Manufacture. The answer may lie in the recovery of essences from vapors given off from vacuum preserve kettles. It is necessary only to replace the jet condensers with surface condensers and to install storage tanks for the condensates. These would then be processed in essence recovery equipment somewhat simpler in design than the arrangement we saw in figure 2. This process is now in commercial use. The aromas can be returned to the preserves to enhance flavor. This practice was referred to recently in the advertisements of a large preserve manufacturer. Sometimes the essences are sold to flavor other food products; for example, milk drinks, ice cream, or candy. The condensates accumulated from a few days' operation in a preserve plant can provide enough essence to permit extensive composition studies. Advantage has been taken of this fact by several groups studying the composition of strawberry flavor. Naturally, preserve essences partake of the character of both the fresh and cooked fruit.

There is another source for apple essence at least, and that is the juice of peels and cores of sound apples used in making vinegar. Volatile concentrate can be recovered profitably from such juice without impairing the quality of the vinegar, affording the manufacturer another source of income.

Powdered Fruit Juices. If 4-fold fruit concentrates are good and 7-fold fruit concentrates better, why not remove all the water and produce powdered juices? This has been done commercially with citrus juices. More recently a comparatively simple method has been developed on a pilot plant scale at the Philadelphia labo-

ratory. It is applicable to the less heat-sensitive juices such as apple, grape, cherry, and probably others.

Three major problems must be solved if the powder is to yield a good juice on reconstitution. First, the water must be removed under conditions which will not damage the flavor; second, the aroma must be recovered and restored without re-introducing an objectionable amount of water; and third, the product must be noncaking and stable on storage.

The first steps in the process are the same as those for making a full-flavored concentrated juice. That is, the aromas are stripped off and concentrated to an essence of about 150-fold, and the stripped juice is then vacuum concentrated to about 75° Brix. By 150-fold we simply mean that the aroma in the juice has been concentrated to a volume 1/150 of that of the juice. This is about the strength of the fruit essences of commerce. Obviously, it is much too dilute for restoration to a powdered juice as too much water would be introduced. It is necessary to fractionate 150-fold essence to approximately 1,000-fold. This is usually done in an efficient laboratory column as the volumes involved are relatively small.

Using the 75° Brix juice concentrate and 1,000-fold essence, powdered juices are made as shown in figure 4. The concentrate is fed through a metering pump to a preheater where its tempera-

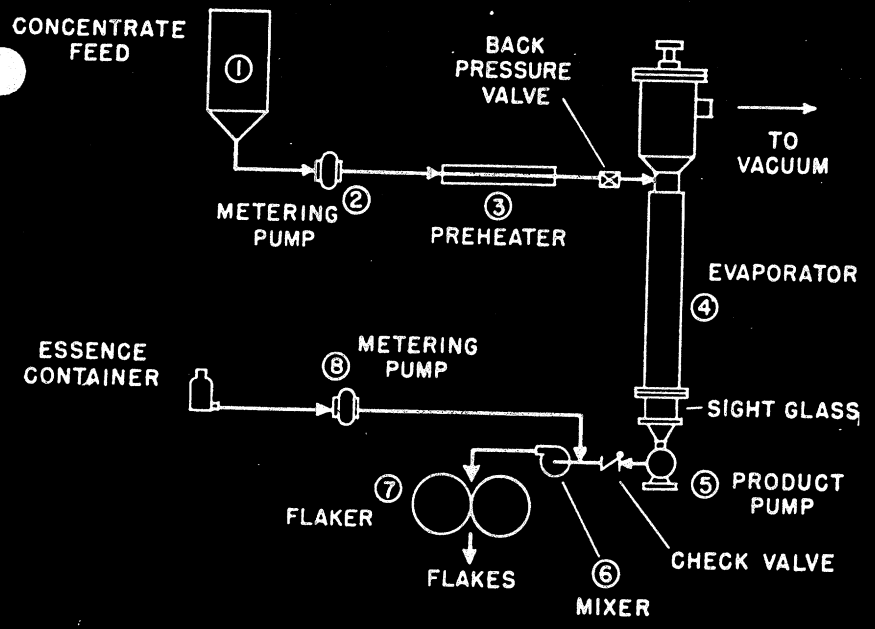

Figure 4. Apparatus for continuous dehydration of fruit juices.

ture is raised to about 135° F. using vacuum steam as the heating medium. Passing through a check valve it enters a vacuum evaporator of special design. The unit has a rotor operating at such a speed that the viscous concentrate is kept in a thin film on the heated walls. With steam of 10 pounds per square inch in the jacket and a vacuum of about 27 inches of mercury, concentration may be carried in a few seconds to approximately 98 percent solids. Under the vacuum mentioned, the product will be at about 230° F. as it leaves the evaporator and, in effect, is a molten powder. It is pumped out by a positive delivery pump. The 1,000-fold essence is now introduced at a metered rate on the positive delivery side of the product pump. Although the temperature of the concentrate is far above the atmospheric boiling point of the essence, the essence does not boil at the point of its introduction as the system is under positive pressure. Immediately following the point of introduction, a mixing device such as a centrifugal pump intimately blends the essence and concentrate and discharges it to flaking rolls. Obviously the distance between the point of discharge from the evaporator and the chilled rolls must be kept at a minimum to avoid heat damage. The flaked product can be easily crushed to a readily soluble powder which on dissolving in cold water yields a fruit juice possessing to a surprising degree the flavor attributes of the fresh fruit juice.

A very small amount of water is inevitably introduced with the essence, but this only raises the moisture content from about 2 percent to 2½ percent. The powders, of course, are very hygroscopic and must be handled in a low humidity atmosphere. If they are packaged with a desiccant, they can be stored for more than a year with practically no change in flavor. Even at 100° F., the flavor stability is excellent and caking will not occur after the desiccant has reduced the moisture to about 1½ percent. This may require several weeks storage at room temperature.

In conclusion I think it can be said that this development of commercially practical methods for aroma recovery has made available concentrated volatile flavors in quantity to permit extensive study of their composition, and it has enabled the production of ice cream, milk drinks, candy, jellies, and other food products possessing fresh fruit flavor. It also may have pointed a way toward concentrated and powdered coffee and tea of better flavor.

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Discussion

QUESTION:

I would like to ask Mr. Eskew if he has had any experience in connection with the Muscatine grapes from the southern part of the country.

ESKEW:

No, our only experience with grapes has been with the Concord grape.

MITCHELL:

I might ask Mr. Eskew about his statement concerning the deliberate attempt to alter flavor of juices by increasing the ester formation. I wonder if you would go into that a little bit more?

ESKEW:

I wish that I could go into it a little more. Unfortunately, the only information that I have on that is very fragmentary. I recently had a talk with this gentleman from Holland, but he did not mention anything that I could add to what I have already said.

The Volatile Flavors of Strawberry[a]

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Interest in the volatile flavor constituents of strawberries reflects the need for objective methods of assaying flavors. In the large scale fruit-breeding programs underway, in the determination of maturity, and especially in processing and in the post-processing history of fruits, we must know what causes the char-

[a] Presented at the Symposium on Chemistry of Natural Food Flavors by Dr. Corse.